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(54) INTERLAYER FILM FOR LAMINATED GLASS AND LAMINATED GLASS

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an interlayer film for laminated glass which has excellent mechanical property, transparency, in particular, haze, thermal insulation and electromagnetic wave transmission and shows excellent resistance to penetration in the lapse of time when being fabricated into a laminated glass and to provide a laminated glass.

SOLUTION: In this interlayer film for laminated glass, a polyvinyl acetal resin, phillosilicate, plasticizer, adhesive power adjusting agent, ITO fine particle and dispersant are included and the ITO fine particle and laminar silicate are dispersed microscopically-uniformly. The laminated glass is also provided.

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CLAIMS

[Claim(s)]

[Claim 1]An interlayer, wherein it contains polyvinyl-acetal resin, a sheet silicate, a plasticizer, an adhesive strength regulator, ITO (tin dope indium oxide) particles, and a dispersing agent and uniform dispersion of ITO particles and the sheet silicate is carried out minutely.

[Olaim 2]Polyvinyl-acetal resin 100 weight section, 0.05 to sheet silicate 20 weight section, At least one or more kinds of 0.0001 to metal salt 1.0 weight sections chosen from a group which consists of 20 to plasticizer 100 weight section, alkali metal salt, and alkaline earth metal salt, The interlayer for glass laminates according to claim 1 consisting of 0.1 to ITO particle 3.0 weight section, and 0.001 to dispersing agent 5.0 weight section, distributing and becoming further so that sheet silicates with a

aspersing agent 3.0 Weight section, distributing and becoming undiet so that since since since is a micrometers or more may be ten or less 100 micrometer² hits.

[Claim 3]Mean particle diameter is 80 nm or less, and ITO particles in a film are the particle numbers of not less than 100 nm. The interlayer for glass laminates according to claim 1 or 2 currently

distributing so that it may become one piece less than/1 micrometer?

[Claim 4]The interlayer for glass laminates according to any one of claims 1 to 3, wherein a sheet silicate is an organicity-ized sheet silicate.

[Claim 5] The interlayer for glass laminates according to any one of claims 1 to 4, wherein a dispersing agent is at least one or more sorts chosen from a group which consists of a phosphoric ester system,

recinoleic acid, and polyrecinoleic acid. [Claim 6]The interlayer for glass laminates according to any one of claims 1 to 5, wherein polyvinyl** acetal resin is polyvinyl butyral resin.

Claim 7]A glass laminate using the interlayer for glass laminates according to any one of claims 1 to

[Glaim 8]The glass laminate according to claim 7 with which Hayes is characterized by visible light transmittance's being not less than 70%, and solar transmittance (300 nm = 2500 nm) being 80% or less of visible light transmittance 1.0% or less.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

aminates and it which were excellent in transparency, thermal insulation nature, and electromagnetic Field of the Invention] This invention relates to the glass laminate which uses the interlayer for glass wave permeability, and adhesive strength with glass is suitable for, and were excellent also in penetration resistance.

Description of the Prior Art]Conventionally, since it is rare for the fragment of glass to disperse and windowpanes, such as a vehicle like a car, an airplane, and a building, etc. Generally what makes the interlayer for glass laminates which consists of polyvinyl-acetal resin, such as polyvinyl butyral resin mentioned glass laminate, makes unify, and is obtained is used. The art of the interlayer in which the although the above conventional glass laminates were excellent in safety, they had the problem of it is safe even if it damages in response to an external shock, the glass laminate is widely used as interlayer excellent in intensity, pliability, transparency, etc. Although the requests to the glass plasticized with the plasticizer, intervene between the glass of a couple at least as the above laminate which was excellent in the thermal insulation nature to which importance was seldom attached until now as a glass laminate for a car or buildings on the other hand were mounting. sheet silicate was minutely distributed in the film is also indicated by JP,2001-58853,A as an being inferior to thermal insulation nature.

processing, etc. The heat ray cut glass etc. which provided coating layers, such as metal or a metallic weak from the outside to the abrasion, and since chemical resistance was also inferior, the method of intercepting the windshield of a car, side glasses, and the infrared rays containing the glass window oxide, in the glass surface, and gave thermal insulation nature are marketed. This coating layer was have the wavelength of not less than 780 nm also in a beam of light bring about a rise in heat, they rays, since a thermal effect is large, and is absorbed by the substance and the infrared rays which 0003]Generally, although an amount of energy is as small as about 10% compared with ultraviolet are called the heat ray. Therefore, in order to improve thermal insulation nature, the method of laminating interlayers, such as a plasticization polyvinyl-butyral-resin film, and using as a glass and glass door of a building is examined, and by for example, vacuum evaporation, sputtering laminate, for example was adopted.

adhesive property of a multilayer coating tip and an interlayer fell, and exfoliation and the white blush 5.8 GHz), satellite broadcasting (12 GHz), etc. are increasingly carried in a car, [7 MHz,] The abovebetween the interlayers for glass laminates is indicated by JP,61–52093,B, JP,64–36442,A public law, example, a ham radio, in recent years. Although urgency traffic apparatus, (10 MHz or less), VICS (a problems, such as interfering with communication functions, such as a cellular phone, car navigation, car information communications system, 2.5 GHz), ETC (a toll road electronic toll collection system, mark of an interlayer happened. They are various kinds of communication equipment (3.5 MHz), for mentioned multilayer coating tip layer checked the penetration of electromagnetic waves, and had a garage opener, and a fee automatic acceptance system. A heat barrier is not provided in a glass (visible light transmittance) falls since it is expensive and the multilayer coating tip is thick, or the plasticization polyvinyl-butyral-resin film, were laminated, There was a problem that transparency surface, but the glass laminate which laminated the polyester film which vapor-deposited metal 0004]However, the heat ray cut glass in which interlayers, such as the above-mentioned

JP,2003-261361,A [DETAILED DESCRIPTION]

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etc. However, there were problems, like exfoliation not only takes place, but as for a glass laminate of the above-mentioned indication, there is a problem in the adhesive property between a plasticization polyvinyl-butyral-resin sheet and polyester film, and the interface of an electromagnetic wave penetration is also insufficient, and it is by it.

especially Hayes are good, and the purpose of this invention is to provide the glass laminate using the Problem(s) to be Solved by the Invention Intensity and pliability are compatible, and transparency, interlayer for glass laminates excellent in thermal insulation nature, and this interlayer for glass aminates, without moreover reducing electromagnetic wave permeability.

agent, and ITO particles and a sheet silicate are the interlayers by which uniform dispersion is carried Means for Solving the Problem This invention contains polyvinyl-acetal resin, a sheet silicate, a plasticizer, an adhesive strength regulator, ITO (tin dope indium oxide) particles, and a dispersing out minutely.

electromagnetic wave permeability were able to be reduced, and thermal insulation nature was able to [0007]In this invention, by using ITO particles, in order to raise thermal insulation nature, and carrying be raised without things. Intensity and pliability were able to be reconciled in this invention, without out uniform dispersion of this ITO particle minutely in a film, transparency (especially Hayes) and reducing transparency (especially Hayes), since a sheet silicate is used and uniform dispersion is minutely carried out into a film.

[0008]If polyvinyl-acetal resin used by this invention is polyvinyl-acetal resin produced by acetalizing 80–99.8–moß of PVA resin is generally used. Although in particular a molecular weight and molecular polymerization of PVA resin used as a raw material, and resin of the degrees of polymerization 500– 2000 is used especially preferably. If the penetration resistance of a glass laminate obtained as the above-mentioned average degree of polymerization is less than 200 falls and the above-mentioned average degree of polymerization exceeds 3000, the moldability of a resin layer worsens, moreover, polyvinyl alcohol (PVA) resin by aldehyde, it will not be limited in particular. The above-mentioned PVA resin is obtained by usually saponifying polyvinyl acetate, and, as for a saponification degree, weight distribution of polyacetal resin that are used for this invention are not restricted, from a [0009]As long as are used, and a carbon number chooses suitably, and is used according to moldability, physical properties, etc., a thing of 200-3000 is preferably used for a degree of the rigidity of a resin layer will become large too much, and processability will worsen.

is mentioned especially. After these acetal resin takes required physical properties into consideration, [0010]As desirable polyvinyl-acetal resin, polyvinyl-butyral (PVB) resin acetalized with butylaldehyde example n-butylaldehyde, isobutyraldehyde, N-valeraldehyde, 2-ethylbutylaldehyde, n-hexylaldehyde, combined two or more kinds of aldehyde at the time of acetalization, and to use suitably. The degree of acetalization of said polyvinyl-acetal resin used by this invention is usually 40 to 85%, and is 60 to n-octyl aldehyde, n-nonyl aldehyde, n-decyl aldehyde, formaldehyde, acetaldehyde, benzaldehyde, etc. are mentioned. As aldehyde used preferably, n−butylaldehyde, n **HEKI sill aldehyde, and n− performance demanded, its **** is good and aldehyde of 1-10 is required for it as the abovementioned aldehyde, two or more kinds may be used together. As an example of aldehyde, for it may be blended in a suitable combination. It is also possible ** polyvinyl- acetal resin which valeraldehyde are mentioned. A carbon number is butylaldehyde of 4 especially preferably.

[0011]A sheet silicate used by this invention has an exchangeable cation between layers of a detailed flaky crystal about 1 nm thick, In [are the silicate mineral condensed in layers by an ionic bond, and] held and also a function as an inorganic bulking agent, a filler, and a viscosity controlling agent can be flake uniformly in a transparent resin composition, the transparency of a resin composition would be this invention, The layer structure is exfoliated by a chemical or physical means, by distributing this exhibited in a resin composition.

montmorillonite, There is a vermiculite, halloysite, or swelling mica besides smectite system argillite, such as saponite, hectorite, beidellite, a stevensite, and nontronite, etc., and what was compounded [0012]Although a kind in particular of the above-mentioned sheet silicate is not limited, [a natural thing or] can be used preferably.

[0013]As shape of a sheet silicate used for this invention, 0.01-3 micrometers and thickness are

used for average length, a thing of 20–500 is preferably used for 0.001–1 micrometer and an aspect ratio, more suitably, 0.05–2 micrometers and thickness are used for average length, and a thing of 50–200 is used for 0.01–0.5 micrometer and an aspect ratio.

silicate to become firm, and for INTAKARE by plasticizer and a cation system surface-active agent to [0014] Although cation exchange capacity in particular of a sheet silicate used for this invention is not organicity-ized sheet silicate, it is satisfactory in any way. Since between layers of a sheet silicate is limited, it is preferred that it is 50 to 200 mmol/100g. In a case below 50 mmol(s)/100g, quantity of a easy to be distributed more minutely in resin than a sheet silicate as for which organicity-ization is a 0015]Athough a sheet silicate may be used as it is and an organicity⊣ized sheet silicate organicity─ surface-active agent, the above-mentioned organicity-ized sheet silicate is used more suitably. As a cation system surface-active agent used for organicity-ization, quarternary ammonium salt, the 4th disteary(dimethylbenzylammonium salt, a Jl hardening beef tallow dimethylannmonium salt, disteary) class phosphonium salt, etc. are mentioned, and quarternary ammonium salt which has at least one with a carbon numbers of eight or more alkyl chain preferably is used. When it does not contain an ammonium salt which has a with a carbon numbers of eight or more alkyl chain, For example, lauryl between crystal layers decreases easily, and a sheet silicate may not be distributed minutely as a ization∼processed beforehand may be used, it is preferred to use an organicity−ized sheet silicate. Under the present circumstances, even if a sheet silicate which is not organicity-ized exists in an cannot be strong, and cannot fully un-polarize between layers of a sheet silicate. As quartemary alkyl chain whose oarbon number is eight or more, the hydrophilic nature of alkyl ammonium ion result. On the other hand, it may be difficult for associative strength between layers of a sheet sheet silicate which it comes to process and which is not organicity-ized with a cation system plasticizer and a cation system surface-active agent which are intercalated by ionic exchange become insufficient, and to distribute a sheet silicate minutely, in exceeding 200 mmol/100 g. trimethylammonium salt, a stearyl trimethyl AMMONIMU salt, trioctyl ammonium salt, a dibenzylammonium salt, etc. are mentioned.

[0016]As for an addition of a sheet silicate, it is preferred that they are 0.05 to 20 weight sections to polyvinyl-acetal resin 100 weight section. In less than 0.05 weight sections, when it is few, and an addition does not fully come to demonstrate desired physical properties but adds exceeding 20 weight sections, a pitch which ** occupies in an increase composite material may decrease, a fall of physical properties, such as a fall of transparency, aggravation of Hayes, and shock resistance, etc. may be produced, and it is not desirable. An addition of a more desirable sheet silicate is 0.5 to 5 weight section.

[0017]A sheet silicate needs to distribute minutely and it is not preferred on mechanical strength, especially transparency that many sheet silicates with a viewing and a size of 1 micrometers or more which can be checked on a scanning electron microscope (SEM) level exist as a grade of the distribution. Quantity of a sheet silicate of 1 micrometers or more or an organicity-ized sheet silicate is ten or less per 10 micrometers x 10 micrometers, and a desirable dispersion state is five or less pieces still more preferably.

[0018]Especially as a method of distributing a sheet silicate minutely in a film, although not limited, especially a thing for which a sheet silicate and a plasticizer are mixed beforehand, and what fully swelled an interlayer spacing of a sheet silicate is added and kneaded to resin is preferred. By mixing a sheet silicate and a plasticizer beforehand, it is because the time of a plasticizer swelling and said sheet silicate being mixed with resin becomes is easy to be distributed in resin minutely easily. In this case, some plasticizers and the whole quantity of a sheet silicate are once mixed, and a residue of a plasticizer may be applied further after that and it may mix.

[0019]As a plasticizer, especially if conventionally used for an interlayer or polyvinyl-acetal resin, it is not limited, and phosphoric acid system plasticizers, such as organic acid ester system plasticizers, such as monobasic—organic-acid ester and polybasic organic acid ester, an organic phosphorus acid system, and an organic phosphorus acid system, etc. are used, for example. As a monobasic—organic-acid ester system plasticizer, for example Triethylene glycol, Glycol and butanoic acid, such as tetraethylene glycol and tripropylene glycol, Isobutyric acid, caproic acid, 2-ethylbutanoic acid, heptylacid, n—octylic acid, Glycol system ester obtained by a reaction with monobasic organic acid, such as 2-ethylhexyl acid, pelargonic acid (n—nonylic acid), and decylacid, is mentioned, Triethylene glycol di-2-ethylbutylate especially, Monobasic—organic—ex*** prong acid ester, triethylene glycol di-2-ethylbutylate especially, Monobasic—organic—

acid ester of triethylene glycols, such as triethylene glycol di-n-octyl acid ester and triethylene glycol di-2-ethylhexyl acid ester, is used suitably. As a polybasic organic acid ester system plasticizer, For example, straight chain shape of polybasic organic acid, such as adipic acid, sebacic acid, and azelaic acid, and the carbon numbers 4-8 or ester with letter alcohol of branching is

suitably. As an organic phosphorus acid system plasticizer, tributoxyethyl phosphate, isodecyl phenyl

phosphate, triisopropyl phosphate, etc. are mentioned, for example. One kind is used independently,

mentioned, Especially, dibutyl sebacic acid ester, dioctylazelate, dibutylcarbitol adipate, etc, are used

**** of the above-mentioned plasticizer is also good, and two or more kinds may be used together. [0020]As an example of a plasticizer used especially preferably. For example, triethylene glycol —*****
prong acid ester, triethylene glycol di-2-ethylbutylate, triethylene glycol di-2-ethylhexyl acid ester, etc. are mentioned. These plasticizers are properly used in consideration of compatibility with resin, etc. according to a kind of polyvinyl-acetal resin. [0021]An addition of a plasticizer has 20 to 100 preferred weight section to polyvinyl-acetal resin. 100 weight section. Less than 20 weight sections are insufficient for distributing a sheet silicate minutely, and penetration resistance may fall. When a plasticizer is added exceeding 100 weight sections, bleed out of a plasticizer arises and there is a possibility that optical strain of a glass laminate obtained by the transparency of a resin layer and an adhesive property falling may become large. An addition of a desirable plasticizer is 30 to 60 weight section.

[0022]At least one or more kinds of metal salt chosen from a group of this invention which makes it meet and becomes an interlayer for glass from alkali metal salt and alkaline earth metal salt as an adhesive strength regulator is used. Especially as the above—mentioned metal, it is not limited, for example, sodium, potassium, magnesium, etc. are mentioned. As acid which constitutes the above—mentioned salt, inorganic acid, such as organic acid, such as octylic acid, hexylacid, butanoic acid, acetic acid, and formic acid, or chloride, and nitric acid, is mentioned, and a salt of organic acid, aceton numbers 2–16 is used suitably especially. As still more desirable metal salt, they are carbon numbers 2–16, it is not limited especially as carboxylic acid magnesium salt of the carbon numbers 2–12, or carboxylic acid potassium salt of the carbon numbers 2–12. For example, magnesium acetate, potassium acetate, magnesium propionate, Potassium propionate and magnesium 2-ethylbutanoate and potassium 2-ethylbutanoate and potassium 2-ethylbutanoate and potassium, etc. may be used preferably, and these may be used independently, or two or more sorts may be used together.

[0023]0.0001 to 1.0 weight section is desirable still more preferred to polyvinyl-acetal resin 100 weight section, and an addition of at least one or more kinds of metal salt chosen from a group which consists of the above-mentioned alkali metal salt and alkaline earth metal salt is 0.01 to 0.2 weight section. In less than 0.0001 weight sections, an adhesive strength fall of a periphery takes place under a high humidity atmosphere, and if 1.0 weight sections are exceeded, adhesive strength will become low too much, and also a problem that membranous transparency falls will arise. [0024]In this invention, in order to give thermal insulation nature, ITO particles are used. It is not limited that what is necessary is [especially] just the ITO particles. As particle diameter of ITO particles, 100 mm or less has preferred mean particle diameter of a primary particle. When it exceeds 100 nm, transparency may fall.

[0025]Content of ITO particles has 0.1 to 3.0 preferred weight section to polyvinyl-acetal resin 100 weight section. Content becomes difficult to come out of an infrared cutting effect in less than 0.1 weight sections, thermal insulation nature will fall, if 3.0 weight sections are exceeded conversely, the permeability of visible light will fall, and Hayes will also become large.

[0026]It is necessary to carry out uniform dispersion of the ITO particles minutely into an interlayer, and when an ITO particle kind is not distributing uniformly minutely, transparency (especially Hayes) will fall. It is preferred to distribute that mean particle diameter is 80 nm or less, and a particle number of not less than 100 nm is below one piece/1micrometer² as a dispersion state in inside of an interlayer of ITO particles.

[0027]it is not limited especially as a method of distributing ITO particles in resin, but after making it susually distribute uniformly in carrier fluid which consists of organic solvents, it is made to distribute in resin and uses, but in this invention, it is preferred to make it distribute as carrier fluid using a

plasticizer used for an interlayer and a plasticizer of the same kind.

may be beforehand added by carrier fluid and a dispersing agent may be used for it, when mixing ITO resin, a dispersing agent is used. Uniform dispersion of the ITO particles can be minutely carried out into a film, and membranous Hayes can be made to improve further by using a dispersing agent. It 0028]In this invention, in order to carry out uniform dispersion of the ITO particles minutely into particles with resin, it may be added, and it may be used.

polyoxyethylene-alkyl-ether phosphoric acid, etc. are mentioned, for example. As a compound which phosphorio ester system compound, alkyl phosphate, a polyoxyethylene alkylphenol ether phosphate, has at least one or more carboxyl groups are suitably used as a dispersing agent among these. As a more carboxyl groups, are mentioned. A phosphoric ester system compound and a compound which has at least one or more carboxyl groups, hydroxy acid especially recinoleic acid, and polyrecinoleic system compound, carboxylate, a polyhydric alcohol type surface-active agent, and at least one or [0029] As a dispersing agent, dispersing agents generally used as a dispersing agent of inorganic particles, such as a compound which has a sulfate ester system compound, a phosphoric ester acid are used suitably.

[0030]0.001 to 5.0 weight section is desirable still more preferred to polyvinyl-acetal resin 100 weight production and glass laminate creation, and may foam, or adhesive strength of an interlayer and glass section, and quantity of the above-mentioned dispersing agent is 0.005 to 3.0 weight section, when expect but exceeds 5.0 weight sections, there is a possibility that it may be alike at the time of film quantity of a dispersing agent is less than 0.001 weight sections, and the addition effect can hardly may be improved too much.

condensation of ITO particles that Hayes improves. As an addition of the above-mentioned chelating dispersing agent, and a compound which has at least one or more carboxyl groups except the above— [0032]As a compound with one or more carboxyl groups except the above-mentioned recinoleic acid especially as the above-mentioned chelating agent, What has good compatibility with a plasticizer or methane, are desirable still more preferred also especially in a chelating agent, and an acetylacetone agent, 0.001 - the amount part of duplexs are preferred to polyvinyl-acetal resin 100 weight section, and polyrecinoleic acid, Aliphatic carboxylic acid, aliphatic dicarboxylic acid, aromatic carboxylic acid, aromatic dicarboxylic acid, hydroxy acid, etc. are mentioned, and benzoic acid, phthalic acid, salicylic acid, propionio acid, n-butanoic acid, 2-ethylbutanoic acid, n-hexanoic acid, 2-ethylhexanoic acid, nresin is preferred, beta diketones, such as an acetylacetone, benzoyltrifluoroacetone, and dipivaloyl mentioned recinoleic acid and polyrecinoleic acid as a distributed auxiliary agent, a chelating agent, acid, etc. can specifically be used. Especially, aliphatic carboxylic acid of C2~C18 and hydroxy acid is used suitably. When these chelating agents configurate to ITO particles, it is thought by barring there is a possibility of foaming at the time of film production, or producing foaming at the time of are used suitably, and are aliphatic carboxylic acid of C2-C10 more preferably. Specifically, acetic and it is 0.01 to 1 weight section more preferably. When the amount part of duplexs is exceeded, 0031]When distributing ITO particles, a distributed auxiliary agent may be used in addition to a etc. are mentioned. Although it is possible not to limit and to use EDTA and beta-diketones glass laminate production. An effect can hardly be expected to be less than 0.001 copy. octanoic acid, etc. are mentioned.

resin 100 weight section, and a more desirable addition is 0.01 to 1 weight section. When the amount adhesive strength of glass and a film, and the addition effect is hard to accept to be less than 0.001 [0033]As a quantity of a compound with one or more carboxyl groups except the above-mentioned recinoleic acid and polyrecinoleic acid, it is 0.001 - the amount part of duplexs to polyvinyl-acetal part of duplexs is exceeded, there are fear of membranous yellowing and a possibility of spoiling

[0034] Additive agents, such as an antioxidant, an ultraviolet ray absorbent, lubricant, fire retardant, a agent, and a heat ray absorbent, may be added by interlayer for glass laminates of this invention if spray for preventing static electricity, an adhesive strength regulator, ******, a heat ray reflective needed in the range which does not check an effect of the invention. [0035]aithough thiokness in particular of an interlayer for glass laminates of this invention is not what Improvement in penetration resistance, etc. may laminate and use the interlayers for glass laminates is limited — as a glass laminate — the minimum — when required penetration resistance and weatherability are taken into consideration, it is preferred that it is 0.3-0.8 mm practical.

JP,2003-261361,A [DETAILED DESCRIPTION]

currently generally used can be used. Although usual glass may be used as glass, heat absorbing glass rays can be intercepted and the thermal insulation effect can be heightened. As glass used as a glass side and comparatively smaller than 1300 nm in a wavelength area (900 nm - 1300 nm), By combining with glass which absorbs infrared rays of a wavelength band (900 nm - 1300 nm), wide range infrared of this invention or an interlayer for glass laminates of this invention, and other interlayers if needed. ,0036]Although obtained by an interlayer of this invention laminating the above-mentioned interlayer which intercepts infrared rays of a wavelength area (900 nm - 1300 nm) is preferred, and especially less is preferred. Since infrared cutoff performance of ITO particles is large at the long wavelength neat absorbing glass whose transmissivity in a full wave length field (900 nm – 1300 nm) is 65% or aminate, what is called organic glass, such as polycarbonate, polymethylmethacrylate, etc. which for glass laminates, and glass, especially as glass, it is not limited but transparent plate glass were excellent in transparency in addition to inorganic glass, may be used.

distributed in a plasticizer is added and kneaded to resin is preferred. by mixing a sheet silicate and a once mixed, and a residue of a plasticizer may be applied further after that and it may mix. As point ** also of the ITO particles was carried out, adding, after carrier fluid has distributed beforehand is adhesive strength modifier, ITO particles, and a dispersing agent, and just to produce a film. Under silicate spreads and it mixes with resin, easily, into resin, it distributes minutely and burns — it is glass laminates of this invention, to knead polyvinyl-acetal resin, a sheet silicate, a plasticizer, an because it becomes. In this oase, some plasticizers and the whole quantity of a sheet silicate are 0037]What is necessary is not to be limited especially as a method of obtaining an interlayer for plasticizer beforehand, when a plasticizer swells in said sheet silicate, between layers of a sheet the present circumstances, especially thing of a sheet silicate for which what was beforehand

Although it is not limited especially as a method of fabricating an interlayer for glass laminates of this the pressing method, etc., it can be more preferably based on an extrusion process by the biaxial said an extrusion machine, plastograph, a kneader, a Banbury mixer, a calendering roll, etc. can be used. It mechanochemical device, a Henschel mixer, a homogenizer, an ultrasonic irradiation machine, etc. are invention but what is necessary is just to produce a film by extrusion process, the calendar method, plasticizer, an adhesive strength modifier, ITO particles, and a dispersing agent is not limited, either, [0038]Although a device in particular that mixes a plasticizer, a sheet silicate and ITO particles, a generally used. Although a device used for kneading of polyvinyl-acetal resin, a sheet silicate, a is preferred to use an extrusion machine from a viewpoint of producing continuously especially. plasticizer, and a dispersing agent is not limited, planet type agitating equipment, a wet direction, and Hayes can be made to improve further.

transparency, and if solar transmittance (300–2500 nm) is 80% or less of visible light transmittance, it transmitted light is a light whose wavelength is 300~2500 nm here. if visible light transmittance is not has the thermal insulation nature which was excellent since transmissivity of light of an infrared area transmittance are not less than 70%, and Hayes is [solar transmittance] 80% or less of visible light transmittance. Visible light refers to light whose wavelength is 380-780 nm, and the solar radiation [0039]As for a glass laminate of this invention, it is preferred that 1.0% or less and visible light less than 70% it comes out and there is Hayes of a glass laminate at 1.0% or less, it excels in by the side of long wavelength decreased rather than visible light.

for example, as functional glass laminates, such as an insulation glass laminate which gave insulation etc. An interlayer for glass laminates of this invention is laminated with rigid bodies other than glass, conveniently for a glass part of vehicles, such as a windshield of a car and side glasses, an airplane, and a train, structural glass, etc. It laminates with other films and it is also possible by using to use for example, metal, an inorganic material, etc., and application as a damping raw material is also [0040] A glass laminate using an interlayer for glass laminates of this invention can be used

and decline in visible light transmittance, aggravation of Hayes, etc. pose a problem. However, since it [0041](OPERATION) When equivalent to a visible light wavelength or high visible light transmittance interlayer like a sheet silicate of bulk, with an additive agent, visible light is scattered about strongly is possible in this invention to distribute a sheet silicate minutely efficiently when a plasticizer usually adds an additive agent of size beyond it to resin of transparency like an indispensable nvades between layers when distributing a sheet silicate in resin as mentioned above, Since

was excellent in transparency, and reconciled mechanical-strength intensity and pliability is obtained, (coexistence of mechanical-strength intensity and pliability) of an interlayer of became possible, and transparency is secured and it is not necessary to remove a plasticizer, an interlayer for glass laminates of outstanding physical properties is obtained easily. That is, an interlayer for glass laminates which is the purpose of original inorganic substance addition and which refining with high visible light transmittance maintained.

it distributes uniformly in the state of a nano-scale ultrafine particle, and dispersion is not caused, an coating which were conventionally used for a thermal insulation nature interlayer, When reflection of a were minutaly distributed by interlayer. Since ΠO particles are smaller than visible light enough since communication functions, such as a cellular phone, car navigation, and a garage opener, it does not minutely distributed in an interlayer with a gestalt of an ultrafine particle, It differs from **** glass obtained, holding transparency, since ITO particles which moreover have infrared absorption ability ultrafine particle, it can process like a manufacturing method of the usual interlayer, processability, [0042]On the other hand, it is transparent, and an interlayer with the thermal insulation effect is and a heat ray reflective glass laminate using heat ray reflective PET by vacuum evaporation or interlayer excellent in transparency, especially a haze value is obtained. Since ITO particles are communication wavelength belt does not take place but it is considered as a glass laminate, to become a problem at all. By distributing a plasticizer beforehand and furthermore, using an ITO workability, productivity, etc. are not spoiled, and it is obtained as usual.

[0043]The more a sheet silicate generally distributes a sheet silicate minutely in resin as a secondary sheet silicate with a nano-scale as mentioned above, it becomes possible to give various functions to When an interface product of a sheet silicate and resin increases with improvement in distribution of thermoplastics-sheet silicate composite, gas barrier property, and transparency improve remarkably. distributed degree of a sheet silicate improves, since dynamics intensity, such as an elastic modulus distributed degree of a sheet silicate improves. By composite-izing an ITO ultrafine particle and a of polymer, increases by restraining molecular motion of polymer in an interface of resin and an inorganic substance, when a gas molecule diffuses inside of a composite material, it is spread bypassing an inorganic substance. Therefore, a gas barrier can be efficiently raised, so that a inorganic crystal. Since a resin layer tends to diffuse a gas molecule far as compared with an a sheet silicate can explain. That is, polymer intensity can be efficiently increased, so that a effect with addition sweet red bean soup with mochi, the more a mechanical strength of a an interlayer.

comparative example.

Example Hereafter, the contents of this invention are explained based on an example and a

hold the system of reaction at 60 ** for 3 hours, and the reaction was made to complete, superfluous and dissolving of 275 g of the PVA resin (average-degree-of-polymerization 1700 and saponification hours with still more superfluous water. The degree of formation of average butyral of this resin was chloride catalyst, and white powdered PVB resin was obtained through rinsing and desiccation for 2 degree % of 99.2 mol) were added and carried out to the pure water 2890g. Temperature control of Examples 1-9, the comparative examples 1-4, 6-7 [Composition of a polyvinyl butyral] The heating the system of reaction was carried out to 15 **, 35% of the weight of the chloride 201g and the nbutylaldehyde 157g were added, this temperature was held, and the reactant was deposited. Then, water washed, unreacted n-butylaldehyde was flushed, sodium hydroxide solution neutralized the

weight section was taught, and ITO particles were distributed in the plasticizer in the level type micro distribution plasticizer was produced. The mean particle diameter of the ITO particles of a distributed bead mill, using phosphoric ester of nonylphenyl polyethylene oxide as a dispersing agent. Then, it HEKISHI rate 20 weight section, ITO powder (mean particle diameter of primary particle: 30 nm) 1 [0045][Production of an ITO distribution plasticizer] As opposed to triethylene glycol di-2-ethyl added under stirring of acetylacetone 0.1 weight section in the solution concerned, and the ITO plasticizer was 35 nm.

Production of a sheet silicate distribution plasticizer] Triethylene glycol di-2-ethyl HEKISHI rate 20 weight section and swelling mica (trade name MAE, CO-OP CHEMICAL GO., LTD. make, organicityzed processing article) I weight section were mixed for I minute with planet type agitating

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equipment, and the paste state sheet silicate distribution plasticizer was obtained.

forming was carried out for 30 minutes at 150 ** using the press-forming machine, and the interlayer quantity of Table 2, The specified quantity of a sheet silicate distribution plasticizer in which a sheet After fully carrying out melt kneading of the specified quantity of 2-ethylbutanoic acid magnesium in of 0.76 mm of average thickness was obtained. The mean particle diameter of the ITO particles in a which magnesium content is furthermore set to 60 ppm to the whole system with a roll mill, press above, the specified quantity of an ITO distribution plasticizer in which ITO particles become the amount of a plasticizer (triethylene glycol di~2~ethyl HEKGSHI rate) becomes 40 weight sections, [0046][Manufacture of the interlayer for glass laminates] PVB resin 100 weight section obtained film is 56 nm, and, as for a not less than 100-nm particle, particle diameter was not observed. A silicate becomes the quantity of Table 2, The specified quantity of the plasticizer that the total sheet silicate of 1 micrometers or more was not observed.

[0047][Manufacture of a glass laminate] The interlayer for glass laminates obtained above is put from the both ends with a transparent float glass (30 cm by 30 cmx2.5 mm in thickness), After putting this 20torr for 20 minutes, it moved to oven, deaerated, and the vacuum press was carried out, holding for 30 minutes at 90 more **. Thus, sticking by pressure was performed for the glass laminate by which in in the rubber back and carrying out indirect desulfurization mind with the degree of vacuum of preparative pressure arrival was carried out for 20 minutes on condition of 135 ** and pressure I,2MPa in autoclave, and the glass laminate was obtained.

comparative example 5 triethylene-glycol di-2-ethyl HEKISHI rate 20 weight section distribute ITO 0048]It carried out like Example 1 except using the ITO distribution plasticizer which made particle 5 weight section.

comparative example 8 triethylene-glycol di-2-ethyl HEKISHI rate 20 weight section distribute sheet It carried out like Example 1 except using the sheet silicate distribution plasticizer which made silicate 23 weight section.

lamination coating of a silver film and the thin film of a metallic oxide was carried out in one sheet of does not contain comparative example 9ITO and a sheet silicate as heat reflective glass with which The glass laminate was produced using the usual interlayer (average 0.76 mm in thickness) which the float glass used when producing a glass laminate.

reflective PET by which lamination coating was carried out, inserted with the transparent float glass By two usual interlayers (average 0.38 mm in thickness) which do not contain comparative example 10ITO and a sheet silicate, the silver film and the thin film of the metallic oxide put heat ray from both ends further, and created the glass laminate.

[0049][Evaluation] The following evaluation was performed about the interlayer for glass laminates and glass laminate which were obtained by the above-mentioned example and the comparative example, and the result was summarized in Tables 2 and 3.

 The transmissivity of 300-2500 nm of a glass laminate is measured using an optical property Naoki spectrophotometer (Shimadzu "UV3100"), it asked for the visible light transmittance (Tv) of 380-780 nm, and the solar transmittance (Ts) of 300-2500 nm by JIS Z 8722 and JIS R3106 (1988). 2) Hayes of the glass laminate was measured based on Hayes JIS K 6714.

particles was photoed and observed. The particle diameter of ITO particles was made into the longest particle diameter of all the ITO particles in 10 miorometers x 10 micrometers of the above-mentioned photographing areas was measured, and it asked for mean particle diameter with a volume conversion 0050]3) The transmission electron microscope (TEM) was used after super-flake production of the dispersion state interlayer in the inside of the film of ITO particles, and the dispersion state of ITO average. It asked for the particulate number with a particle diameter of not less than 100 nm which path of the ITO particles in the photograph obtained by the above-mentioned photography. The exists in the above-mentioned photographing area, and the number of per 1 micrometer² was

(1) A viewing device, a condition and a transmission electron microscope:H-7100FA type (made by Hitachi, Ltd.) – aocelerating voltage: — 100-kV(2) section manufacturing device and ultramicrotome: — вм-илялситs (made by the Leica company)

- Freezing cutting system : REICHERT-NISSEI-FCS (made by the Leica company)

– Knife: DIATOME ULTRA CRYO DRY (made by DIATOME)

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silicate into a sample, using the scanning electron microscope (SEM), the range of 10 micrometers x10 micrometers was observed, and the major axis asked for the particle number of 1 micrometers or 4) By making the interlayer for dispersion state glass laminates in the inside of the film of a sheet

(1) A condition and viewing device and scanning electron microscope.S-3500N (made by Hitachi, Ltd.) more, and asked for the number of per 1 micrometer 2 in arbitrary positions

method of a distant place community), and the rate of reflection loss of the range of 2-26.5 GHz (dB) was measured. In quest of the maximum and the minimum of a loss in the above-mentioned perimeter 600 mm square is stood among one pair for transmit receives of antennas. The spectrum analyzer measurement (electromagnetic wave shielding effect measurement of a neighborhood community). Measure the reflection loss value (dB) of the range of 0.1−10 MHz, and, on the other hand, sample received the electric wave from a radio wave signal generator (electromagnetic wave measuring - Accelerating voltage : 15 kV [0051]5) By the electromagnetic wave permeability KEC method wave number range, it was considered as the electromagnetic wave shielding index.

beforehand, and the pan Meru value was calculated for the result according to Table 1. The adhesive particle diameter of glass was set to 6 mm or less. It judged with the boundary sample which carried was adjusted to it, and it ground until the head struck this with the hammer which is 0.45 kg and the property over the glass of an interlayer is estimated by the pan Meru value, it is so large that a pan 6) The pan Meru value glass laminate was neglected to ~18**0.6 ** temperature for 16 hours, and out grade attachment of the degree of exposure of the film after glass carries out partial avuision Meru value is large, and adhesive strength is small when small. [of adhesive strength with glass]

The high-speed hauling examination of the interlayer was done with the universal testing machine on condition of temperature:23 **, humidity:50%RH, and speed-of-testing:200 mm/min using the Mechanical strength (tensile strength)

8) The humidity test constant temperature/humidity chamber was used and the humidity test of the

dumbbell No. 1 type specimen (JIS-K-6771).

glass laminate was done based on JIS R 3212(1992)"automobile safety glass test-method."

Table 1]

[Table 2] [0053]

	事味 事味						
	動化メベバ	9					
對佛狐	動陸教物試壓運	0					
	(v d W) 雑酸の難ぐ治	7.8E					
事項探シー・))	(4 (4 (4 (4 (4 (4 (4 (4 (4 (4 (4 (4 (4 (1~0					
	(%) メトヘ	8.0					
孙华华张	(%) 本服養持日	5,88					
	可提光層阻率(%)	8.28					
OT I 掛雑代	100mm超粒子数(個人μm²)	00.0					
	(加机) 對子游台平	69					
型嫌化な下ア	(em1/國) 漢子球鹽m1	0.00					
量軌端OT I	(衛星重)	1.0					
新史賴 莊外圖	0.1						
		Ţ					
國化在聯盟泰	(溶量室) 最成						

[Table 3] [0054]

especially Hayes are good, and] for glass laminates which can adjust adhesive strength, and the glass laminate using the interlayer can be provided.

[Translation done.]

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	用地スさいは五神族(1 ※ 1 ※ 1 を 1 を 1 を 1 を 1 を 1 を 1 を 1 を 1 を					*					
	立件	×	×	×	×	x	×	×	. ×	×	×
新時期	動れべくだ	9	9	9	9	9	9	9	9	9	9
	網內對鄉加緊領	0	0	0	0	0	0	0	0	×	Xxxx
	(18 9 M) 鬼類の疑ぐ(6	0.6 g	1,88	£ 62	8.68	₽.6E	8.8.6	Z 9 Z	₽. ΥΥ	9.8 S	0.83
化一/ド脳療器	(SPV) \$\d	I~0	T~0	T~0	T~0	1~0	T~0	1~0	T ~ C	12~48	2 E~E I
到停车光	(%) メト〜	3.0	8.0	3.0	8.0	8.1	3.0	3.0	6.8	9 '0	٥٠٥
	(%) 康熙製練日	2,08	\$.08	₽.87	7.27	9,18	0.88	1.98	8.85	0.74	0.74
	山稻米孫陽本(米)	0.68	6'88	1.88	8.78	8.8	9.28	8.28	1.09	.0.08	0,87
	100 nm超程子数(個人 nm 1	-	_	00.0	00.0	00.8	00'0	0.60		_ ·	-
對猶代OT I	(加 点) 翻干场时平	-	_	0.9	89	103	<i>L</i> 9	69		_	
対徴代なトア	("mu/圖) 減于試話mu [_	0.00	-	00.0	0.00	_	0.0	01.0		_
量啟添OTI	(淑華康)	0.0	0.0	30.0	90.0	0.8	1.0	0.1	0.0	-	
滿點類紅粉	(湘暑度) 量は	0.0	to'0	0.0	1.0	0.1	0.0	10.0	12.0	-	-
		τ	2	8	₽	9	9	Ł	8	gram B	I O as
						4 #	₩ ₹				

[0055]
[Effect of the Invention]According to this invention, by making polyvinyl-acetal resin distribute a sheet silicate minutely, Reconoile intensity and pliability, excel in transparency and thermal insulation nature by carrying out uniform dispersion of the ITO ultrafine particle into a film further, and moreover electromagnetic wave permeability is good, The interlayer [it is cheap, and transparency,

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